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THERMOCOUPLE INTERACTIONS DURING TESTING OF MELT INFILTRATED CERAMIC MATRIX COMPOSITES (POSTPRINT)

G. Ojard, G. Morscher, Y. Gowayed, U. Santhosh, J. Ahmad, R. Miller, and R. John

Pratt & Whitney

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14. ABSTRACT
As high performance ceramic matrix composite systems, such as Melt Infiltrated (MI) SiC/SiC, are being considered for advanced gas turbine engine applications, the characterization of the material becomes more important. A series of test were conducted where Pt and Ni sheathed Pt thermocouples were used to monitor temperature for short and long duration fast fracture, fatigue and creep tests. While it is known that Si forms eutectics with Pt and Ni, this was initially not considered an issue. But since MI SiC/SiC composite achieves much of its performance from the infiltrated phase of Silicon (for high conductivity and low porosity), it was felt that further study of possible interactions of the Si phase has to be considered. Post-test real-time X-ray inspection of the mechanical time-dependent and time-independent testing revealed that the extent of alloying into the sample was greater than anticipated an in some cases extended throughout the entire gage section of the tensile bars. It was concluded that the interactions were limited to the Si Phase of the material and that there was no difference between samples affected by alloying versus those that did not.

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THERMOCOUPLE INTERACTIONS DURING TESTING OF MELT INFILTRATED CERAMIC MATRIX COMPOSITES

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ABSTRACT:

As high performance ceramic matrix composite systems, such as Melt Infiltrated (MI) SiC/SiC, are being considered for advanced gas turbine engine applications, the characterization of the material becomes more important. A series of tests were conducted where Pt and Ni sheathed Pt thermocouples were used to monitor temperature for short and long duration fast fracture, fatigue and creep tests. While it is known that Si forms eutectics with Pt and Ni, this was initially not considered an issue. But since MI SiC/SiC composite achieves much of its performance from the infiltrated phase of Silicon (for high conductivity and low porosity), it was felt that further study of possible interactions of the Si phase has to be considered.

Post-test real-time X-ray inspection of the mechanical time-dependent and time-independent testing revealed that the extent of alloying into the sample was greater than anticipated and in some cases extended throughout the entire gage section of the tensile bars. It was concluded that the interactions were limited to the Si Phase of the material and that there was no difference between samples affected by alloying versus those that did not. Additionally, an experimental approach was taken to limit the extent of the thermocouple interaction. The results of this study and the approach will be presented and discussed.

INTRODUCTION

There is ever increasing interest in Ceramic Matrix Composites (CMCs) due to the fact that mechanical properties are relatively constant with temperatures up to the maximum use temperature [1]. This is shown in the interest of CMCs for extended high temperature use where superalloys are usually considered [2,3].

As characterization of this class of material proceeds to enable such applications, interactions present during the characterization become important. Most mechanical testing uses strain gauges, extensometry and or thermocouples. Strain gauges are normally used at room temperature where interactions can normally be considered to be absent and will not be discussed further. Most extensometers use alumina rods, which can be considered inert for the CMC class of material [4]. In addition, extensometers have point interactions with the specimen minimizing the area of contact. It should be noted that the extensometer could be influenced by the presence of porosity near the contact point during testing that would show up as discontinuities in strain [5] but this paper is focused on impact to the sample and not resulting testing impacts.

A main part of characterization is knowing the temperature at which the characterization is occurring. There are two ways to know the temperature: thermocouples (TC) and optical pyrometry. Both of these approaches were used by the authors in characterization and testing but the vast amount of the work has been with thermocouples. Due to the maximum temperature

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range of interest (typically 1204ºC), the thermocouples used consisted of Pt with or without Ni sheathing. In general CMC testing, there should be no issues with using thermocouples since the materials are usually inert: oxide, silicon carbide or silicon nitride matrix composite systems.

This could be an issue with the high performance CMC system generically referred to as the Melt Infiltrated CMC where a silicon based alloy is melted into the material as part of the matrix to increase through thickness thermal conductivity as well as decrease levels of porosity. Si is known to have eutectic interactions with Pt and Ni as well as other metals. (Pt and Ni are specifically pointed out since they are present in thermocouples at the highest percentages.) In this paper, a series of samples were exposed at elevated temperature for long periods of time under load for characterization purposes: creep and dwell fatigue. After testing, real time X-ray was undertaken on select samples to note if there was interaction or not. In addition, testing was reviewed to see if there was any affect on the test results.

PROCEDURE

Material Description

The material chosen for the study was the Melt Infiltrated SiC/SiC CMC system, which was initially developed under the Enabling Propulsion Materials Program (EPM) and is still under further refinement at NASA-Glenn Research Center (GRC). This material system has been systematically studied at various development periods and the most promising was the 01/01 Melt Infiltrated iBN SiC/SiC (01/01 is indicative of the month and year that development was frozen) [6]. There is a wide set of data from NASA for this system as well as a broad historic database from the material development [7]. This allowed a testing system to be put into place to look for key development properties which would be needed from a modeling effort and would hence leverage existing data generated by NASA-GRC.

The Sylramic® fiber was fabricated by DuPont as a 10 μm diameter stochiometric SiC fiber and bundled into tows of 800 fibers each. The sizing applied was polyvinyl alcohol (PVA). For this study, the four lots of fibers, which were used, were wound on 19 different spools. The tow spools were then woven into a 5 harness satin (HS) balanced weave at 20 ends per inch (EPI). An in-situ Boron Nitride (iBN) treatment was performed on the weave (at NASA-GRC), which created a fine layer of BN on every fiber. The fabric was then laid in graphite tooling to correspond to the final part design (flat plates for this experimental program). All the panels were manufactured from a symmetric cross ply laminate using a total of 8 plies. The graphite tooling has holes to allow the CVI deposition to occur. At this stage, another BN layer was applied. This BN coating was doped with Si to provide better environmental protection of the interface. This was followed by SiC vapor deposition around the tows. Typically, densification is done to about 30% open porosity. SiC particulates are then slurry cast into the material followed by melt infiltration of a Si alloy to arrive at a nearly full density material. The material at this time has less than 2% open porosity. Through this process, 15 panels were fabricated in 3 lots of material. Typical cross sections of this material are shown in Figure 1 showing the material phases.

After fabrication, all the panels were interrogated by pulse echo ultrasound (10 MHz) and film X-ray. There was no indication of any delamination and no gross porosity was noted in the panels. In addition, each panel had 2 tensile bars extracted for witness testing at room

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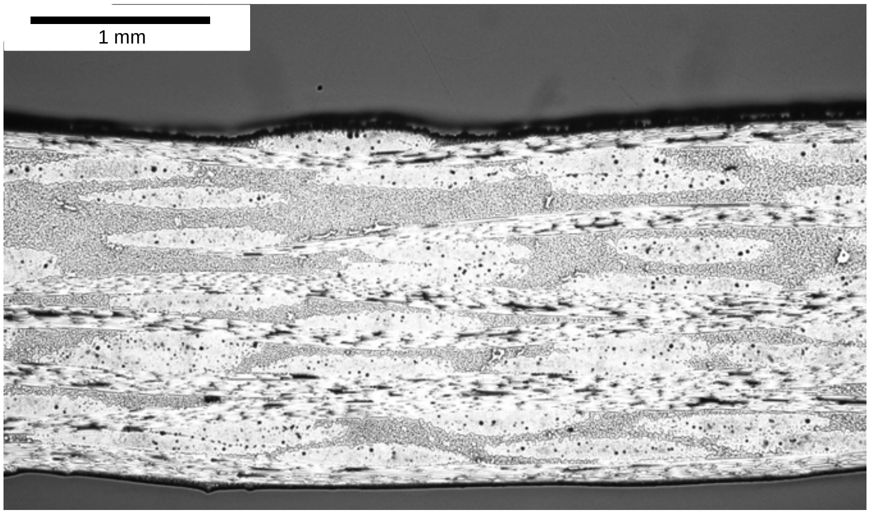
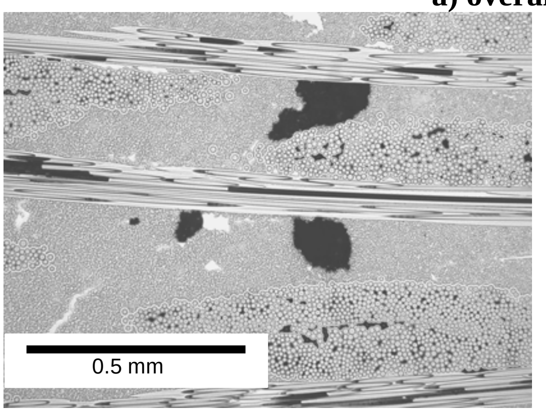
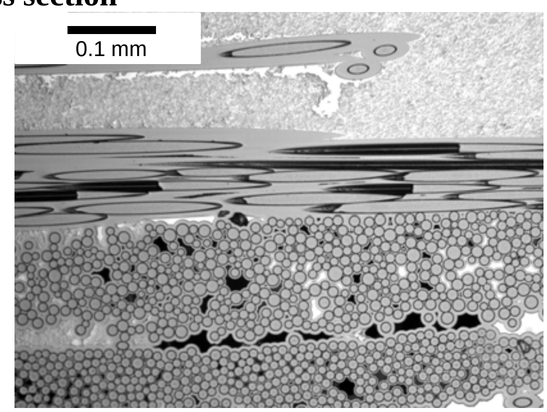
temperature. All samples tested failed above a 0.3% strain to failure requirement. Hence, all panels were accepted into the testing effort.



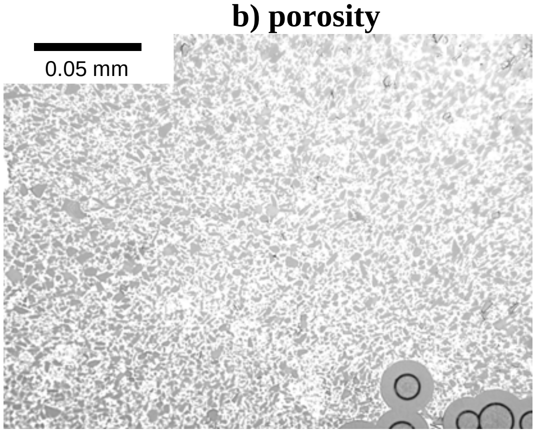
a) overall cross section



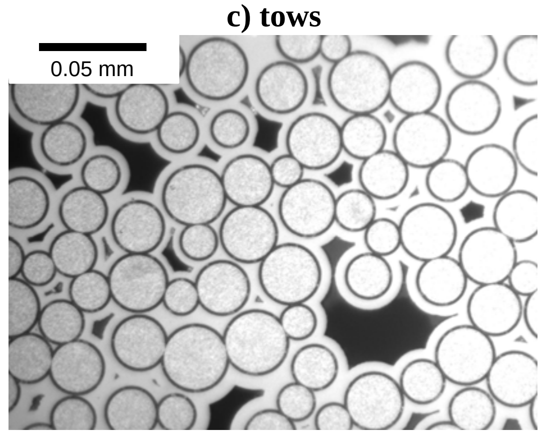
b) porosity



c) tows



d) SiC particulate with Si



e) Interface coating (BN)

Figure 1. MI SiC/SiC Microstructure Images

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Thermocouples

Two types of thermocouples were used to monitor the temperature during testing: Type R and Type K. The vast majority of the testing was done with a Type R thermocouple while the long-term creep testing was done with a Type K thermocouple with the addition of a Ni sheath. A type R thermocouple is a bi-metallic joint between a Platinum –13% Rhodium alloy and Platinum [8]. A type K thermocouple is a bi-metallic joint between a Nickel-Chromium alloy and a Nickel-Aluminum alloy [8]. The thermocouples were held in place by wires.

Mechanical Testing - Durability Testing (Time-dependent)

The mechanical testing of MI SiC/SiC has been reported previously [9,10]. This work has been focused on the long-term durability response of the material under different loads and times: creep and dwell fatigue. Most of the testing was done at 1204ºC with limited testing at 815ºC.

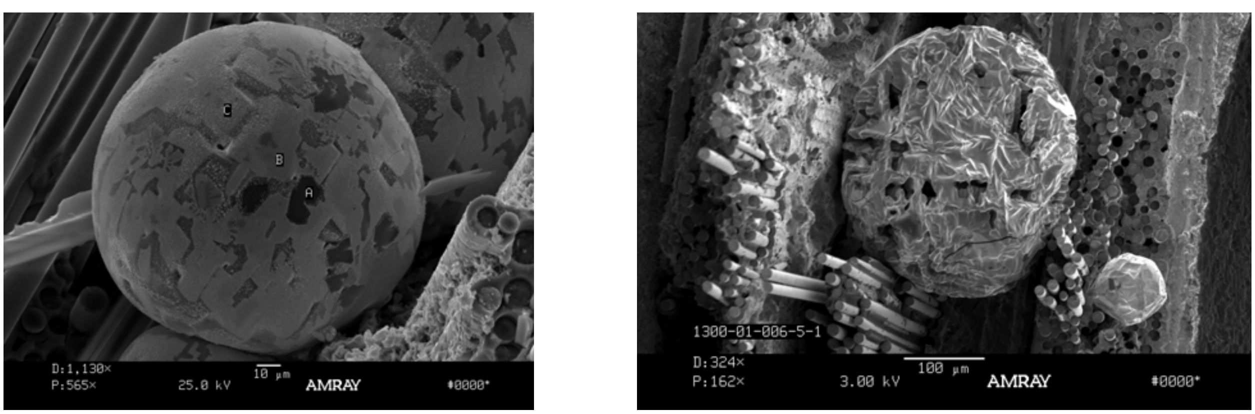
Characterization - Real Time X-ray

As an aid in understanding the material, after creep and dwell fatigue testing, samples were interrogated using a real time x-ray machine. This effort was done trying to understand some low modulus material and was instrumental in finding porosity in the material [11]. In addition, it is capable of determining high-density inclusions in the material. This was one method to determine the presence of alloying occurring during testing.

RESULTS

Determination of initial reactions

During some of the initial testing efforts, thermocouple interactions were noted when Scanning Electron Microscopy (SEM) analysis of the failure face was performed. This is shown in Figure 2 for both the Type R and Type K (with a Ni sheath) thermocouples. With a combination of using Energy Dispersive Spectroscopy (EDS) and consulting binary phase diagrams, it was determined that these were eutectic spheres of Pt-Si (Figure 2a) and Ni-Si (Figure 2b). In addition, since these were at the failure face, it was thought to be a post failure event when the TC slipped onto the fracture face before the furnace was powered off.



a) Type R TC used

b) Type K TC used (with a Ni sheath)

Figure 2. SEM Images of Failure Faces

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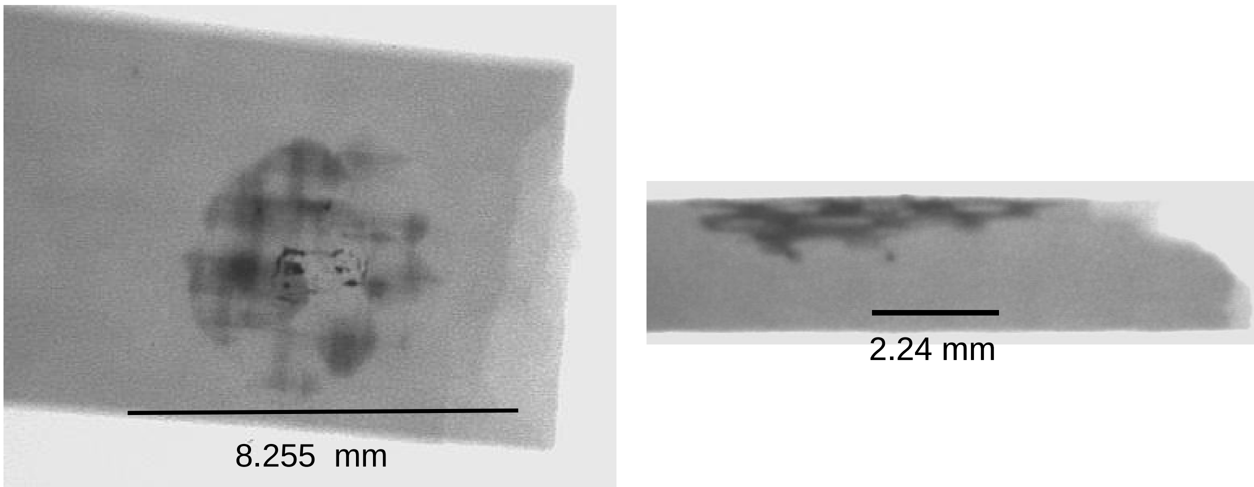
Real Time X-ray Analysis

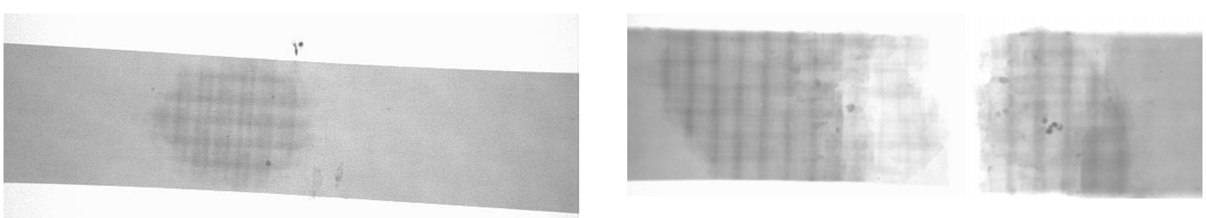
As noted previously, real time X-ray was used to document the presence of porosity in the sample gage region [11]. This was expanded to additional samples as an aid in determining sample outlier status [11]. During interrogation of samples, high-density inclusions were noted in the sample away from the failure face. The resultant real time X-ray images of the sample are shown in Figure 3. The dark inclusions in the material are regions that have been alloyed with Pt during the test. (The contrast is due to the greater absorption of the X-rays from the higher atomic number species present in the sample [12].) This sample was tested at 220.8 MPa for 1.3 hours at 1204ºC suing a Type R TC. Energy Dispersive Spectroscopy (EDS) confirmed the presence of Pt in the SEM.



a) Normal view b) Side View

Figure 3. Real Time X-ray of a Sample showing Interaction
(220.8 MPa creep test at 1204ºC)

This was the first time that the presence of Pt (and in the case for the Ni sheathed Type K TC) was noted in the material and not at the failure face. Based on this finding, past testing was reviewed to note the full extent of the TC-Si interaction. The presence of alloying was greater than anticipated. This is shown in Figure 4 for two creep samples tested at 1204ºC using a Type K TC with a Ni sheath. Figure 4 shows that the interaction zone is not time dependent and it can actually be greater in shorter duration tests.



a) Creep test at 110.4 MPa, 1204ºC, 1269 hours – sample did not fail

a) Creep test at 165.6 MPa, 1204ºC, 477 hours – sample failed (Sample also shown in Fig. 2b)

Figure 4. Real Time X-ray of Two Samples Tested Under Different Stresses

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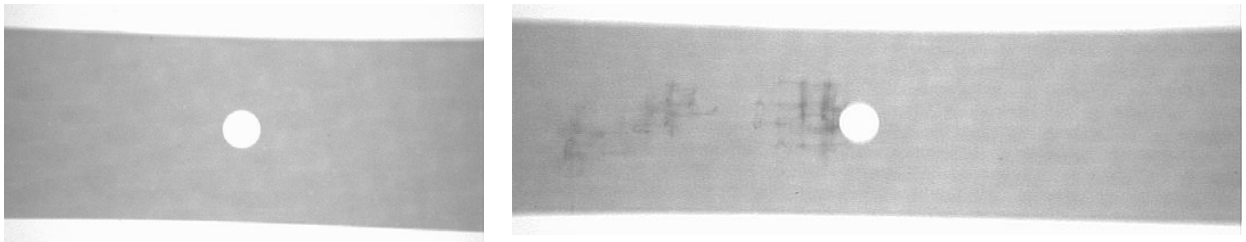
For duplicate tests, the interaction was not always seen. For a series of 1 Hz fatigue tests (110.4 MPa and 1204ºC) with a 2.286 mm through hole, the interaction was seen on one sample and not the other as shown in Figure 5. These samples were tested for 400,000 cycles and neither sample failed. The strain response of the material was identical.



a) No TC interaction b) TC interaction

Figure 5. Real Time X-ray of Two Samples Tested Under Identical Conditions
(1 Hz Fatigue, R = 0.05, 110.4 MPa Peak Stress, 1204ºC)

DISCUSSION

As shown in the results, it is clear that the presence of a thermocouple (or the presence of a metal that can be alloyed with Si such as Pt or Ni) allows diffusion of another metal species into the material. What needs to be determined with confidence is this alloying affecting the mechanical results or not. (Or is the thermocouple affecting the testing outcome?) This was achieved by looking at the mechanical results with the insight gained by knowing if there was or was not any thermocouple interaction (alloying). This review was initially taken by looking at the creep response of some long-term creep durability samples. This is shown in Figure 6 for a series of samples that had a 4.572 mm through hole (W/d for the sample was set at 5) tested at 1204ºC for 1,000 or 2,000 hours under a net section stress of 55.4 MPa.

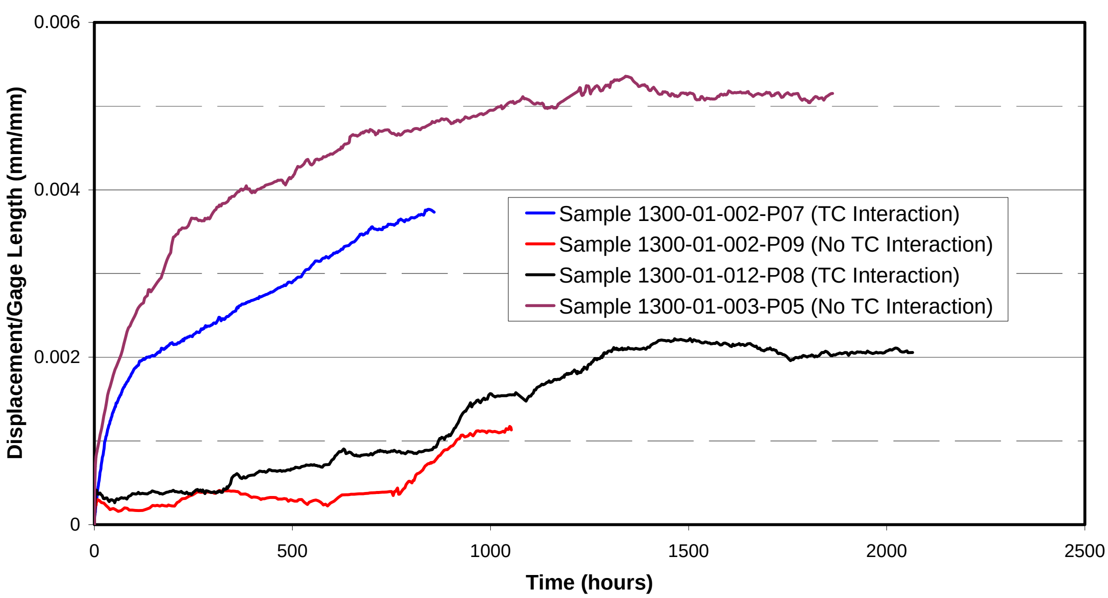


Figure 6. Long-term creep response of samples with and without thermocouple alloying

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As can be seen in Figure 6, there was no differentiation between the displacement/gage length behavior with or without interaction. A review of other testing within the program for standard tensile samples and samples with holes showed similar response in that the presence of alloying did not differentiate the strain or displacement/gage length response. The residual strength testing done on samples that achieved run out backs up this conclusion. Two samples with a 2.286 mm through hole were tested at 110.4 MPa and 1204ºC for 1,000 hours. One sample showed alloying to occur while the other did not. The residual tensile results plus a baseline sample are shown in Figure 7. As can be seen, the tensile curves show no differentiation.

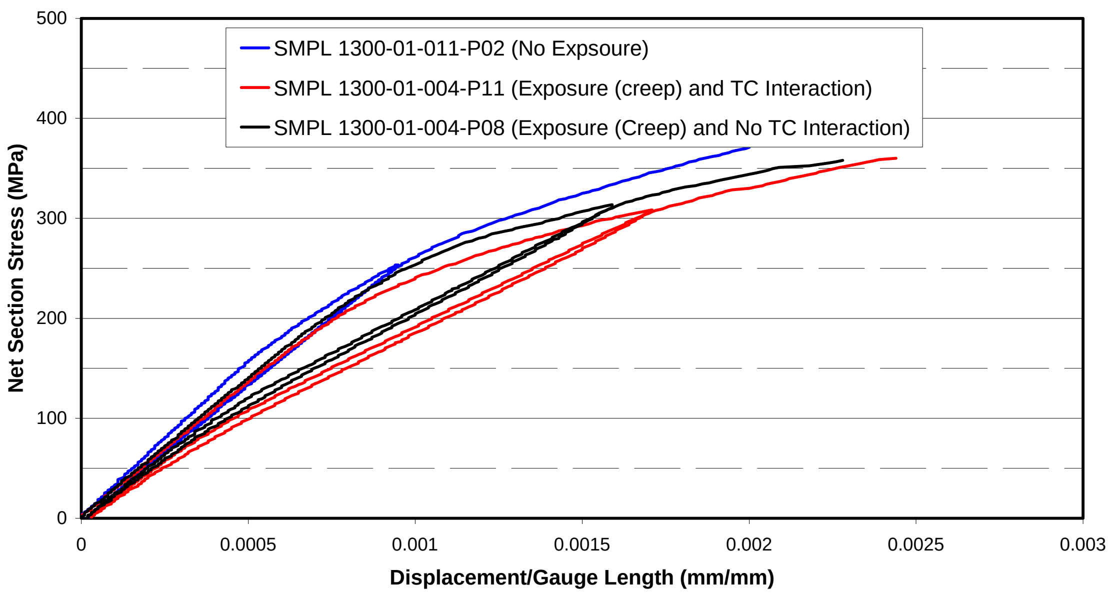

Figure 7. Residual tensile tests of samples with and without thermocouple alloying
(sample with through hole size of 2.286 mm, baseline curve shown for comparison purposes)

In addition, as further indication that there was no effect seen for the alloyed sample shown in Figure 7, posttest analysis showed that the hole dominated the failure location and not the region of alloying. This is shown in a real time X-ray image of the sample after the residual tensile test in Figure 8.

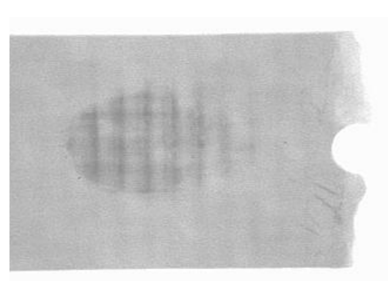

Figure 8. Real time X-ray image of sample with interaction showing that the alloying did not affect the failure location

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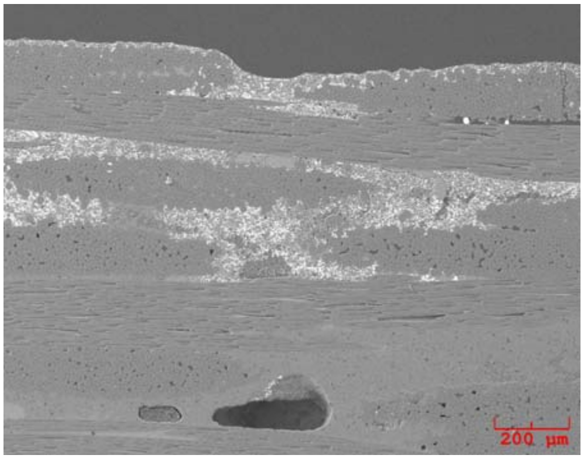
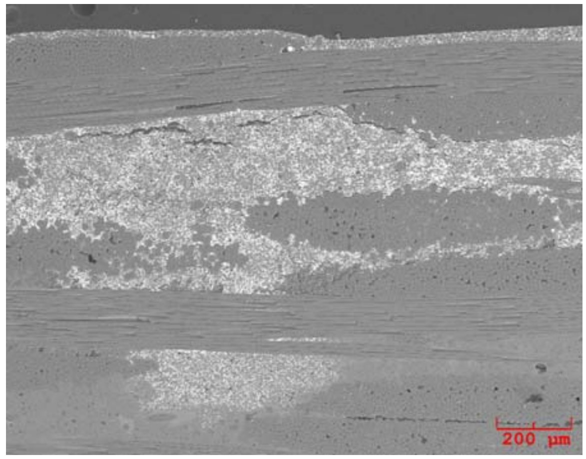
Further analysis was done to note where the alloying was within the cross section of the material. A sample where a Pt thermocouple was used where alloying was known to occur from post real time X-ray analysis was sectioned. Back-scattered images were taken in the SEM and the presence of Pt was highlighted this way. It was seen that the Pt was staying in the melt-infiltrated phase of the material. This can be seen in Figure 9.



a)



b)

Figure 9. Back-scattered SEM image showing regions of Pt (white) alloying occurring within the Si Melt-Infiltrated phase of the CMC

The sample shown in Figure 9 was a creep sample that was tested at 220.8 MPa and 1204ºC and failed at 1.3 hours. The fracture face was probed in the SEM using EDS trying to find the presence of Pt at the failure face. Multiple efforts failed to reveal Pt at the failure face. This is consistent with the real time X-ray image shown in Figure 8 that the thermocouple alloying is not affecting the testing results and or failure location. What is critical in Figure 9 and the back-scattered SEM work done is that it clearly shows that the alloying is not interacting with the CVI SiC phase of the material and additionally is not getting near the fiber interface coating. It would be expected that if the material were able to get to the interface that an effect would be seen.

It appears based on the work done to date that the thermocouple alloying is acting like a marker (tracer) within the system that is staying within the Si phase, which is around the CVI SiC coating of the fiber tows. This is clearly seen in Figure 10 where an edge on view of the sample taken in the real time X-ray machine shows that the alloying is present around the fiber tows. Since the temperature of testing where alloying occurred was always 1204ºC and is above the eutectic point of 979ºC (Si-Pt) and 993ºC (Si-Ni), the alloying phase is highly mobile.

Figure 10. Side view of sample with alloying from real time X-ray
(Creep test at 110.5 MPa and 1204ºC for 1269 hours)

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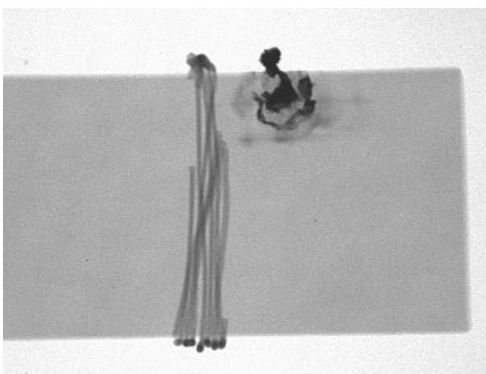
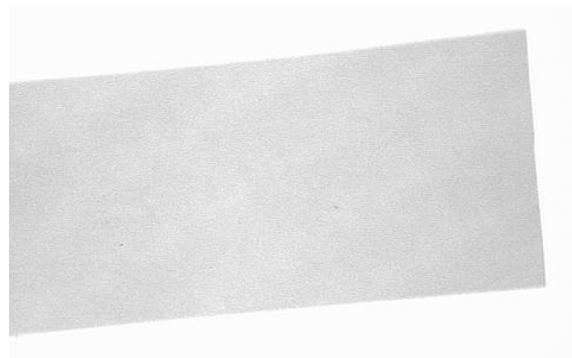
As part of the effort to understand the interaction and to see if the effects can be minimized, a series of experiments were undertaken to note the presence of an interaction (or lack of an interaction) by having thermocouples placed on each side of the panel (from fabrication). As noted previously, the panel is fabricated in one of the last steps by placing Si on one side of the panel and then melting this through the panel. This results in one side of the panel being Si rich and the other side being Si poor. This is mainly seen in the appearance of the panel after fabrication. Thermocouples were placed on scrap material and then isothermally heat-treated at 1093ºC for 12 hours. (While this was not the temperature used for testing, it was above the eutectic points as noted previously.) This was done for 3 samples of each condition: thermocouple on Si rich face and thermocouple on Si poor face. It was found that by placing the sample on the Si rich face that the interaction occurred on 2/3 of the samples while the thermocouples placed on the Si poor face had no interactions. The results of this experiment are shown in Figure 10 for the two cases studied.





a) thermocouple on Si rich face b) thermocouple on Si poor face

Figure 10. Results of interrogating the two sides of the panel showing that the reaction can be seen when the thermocouple is placed on the Si rich face

CONCLUSION

It was seen that the thermocouple did interact with the Si phase of the Melt Infiltrated SiC/SiC composite during testing. Considering that the standard thermocouple materials form eutectics with Si, this is no longer surprising. Reviewing the data generated with insight from X-ray interrogation of the samples, it was seen that the presence of the alloying within regions of the material were not affecting the results. Additionally, there was no effect seen on the failure location of the material. This is further confirmed by the investigative SEM work done where EDS and back-scattered images show that the alloying is staying strictly within the Si phase of the CMC and is not penetrating the CVI SiC around the fiber tows and not attacking the fiber interface coating.

After the interaction was noted, the experimental work done by placing the thermocouples on the two different sides of the panels shows that the interaction can be reduced. By placing the thermocouple on the Si poor side of the panel, the presence of the interaction can be reduced. This should eliminate this as a concern but even with this, care should be taken to look at samples to assure that alloying still has not occurred since the limited testing here could not cover all the conditions that could exist on the Si poor face of the material (there could be local spots on the Si poor face that due to the panel fabrication could be rich in Si).

Corrosion, Wear, Fatigue, and Reliability of Ceramics, Ceramic Engineering and Science Proceedings (CESP), Volume 29, Issue 3, Jonathan Salem and Edwin R. Fuller, editors; Tatsuki Ohji and Andrew Wereszczak, volume editors, pp. 11-20, American Ceramic Society, Wiley, 2008. Presented at 32nd International Conference on Advanced Ceramics and Composites, January 27-February 1, 2008, Daytona Beach, Florida.

All of this work to date is for isothermal testing above the eutectic point. Micro-structural examination upon sample cooling did not show any eutectic phases formed considering the extent of the alloying seen in the real time X-ray. This may be due to the fact that sample cooling after testing was sufficiently fast such that the kinetics of formation of the phases could not occur. It is unknown at this time what would occur if the temperature were held below the eutectic for a sufficient time to allow the phases of the eutectic to form. Thermal cycling with alloying occurring that goes through the eutectic point may be a problem if the phases formed have different volumes. This is an area for concern in use and testing that should be studied. In the end, researchers should be aware of possible interactions when investigating this class of material (MI SiC/SiC) and take care in evaluating their results regardless of how the test is performed (such as the isothermal work presented here).

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